PCT / IN 04 / 0 0 1 4 2

THE PATENTS ACT, 1970

It is hereby certified that annexed hereto is a true copy of PCT International application filed on 19.03.2004 of the extract of PCT Application No. PCT/IN04/00064 by M/S PHARMED MEDICARE PRIVATE LIMITED, Pharmed Gardens, Whitefield Road, Bangalore-560 048,Karnataka,India.

.....

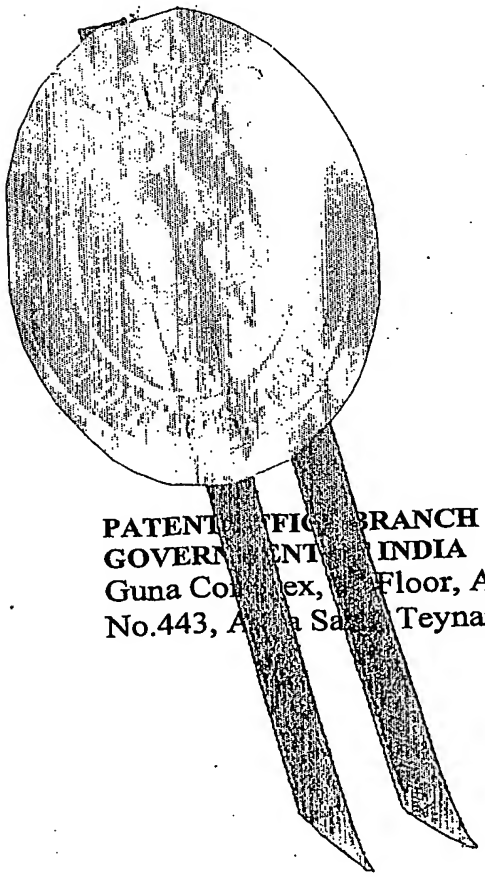
.....In witness thereof

I have hereunto set my hand

Dated this the 14th day of October, 2004
22nd day of Asvina, 1926 (Saka)

M.S.V

(M.S.VENKATARAMAN)
ASSISTANT CONTROLLER OF PATENTS & DESIGNS



PATENT OFFICE BRANCH
GOVERNMENT OF INDIA
Guna Complex, II Floor, Annex.II
No.443, Anna Salai, Teynampet, Chennai – 600 018.

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PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No. PCT/ IN04/00064

International Filing Date 19 MARCH 2004 (19.03.04)

THE PATENT OFFICE, (INDIA)
PCT INTERNATIONAL APPLICATION
Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference (if desired) (12 characters maximum) IN/PA-62

Box No. I TITLE OF INVENTION AN IMPROVED PROCESS FOR PRODUCING CHLORINATED SUCROSE

Box No. II APPLICANT ☐ This person is also inventor

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Pharmed Medicare Private Limited,
Pharmed Gardens,
Whitefield Road,
Bangalore - 560 048, Karnataka State, India.

Telephone No. 91-80-28410158
Facsimile No. 91-80-28410177
Teleprinter No.
Applicant's registration No. with the Office

State *(that is, country)* of nationality: IN
State *(that is, country)* of residence: IN

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Rakesh Ratnam,
No.488, 2/B, 39th Cross,
8th Main, Jayanagar,
Bangalore-560 082, Karnataka State, India.

This person is:
☐ applicant only
☒ applicant and inventor
☐ inventor only *(If this check-box is marked, do not fill in below.)*

Applicant's registration No. with the Office

State *(that is, country)* of nationality: IN
State *(that is, country)* of residence: IN

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

MRS. ALAMELU VAIDYANATHAN,
45, 2nd Cross, 3rd Block, 3rd Stage,
Basaveshwaranagar,
Bangalore - 560 079, Karnataka, India.

Telephone No. 91-80-23235021
Facsimile No. 91-80-23235854
Teleprinter No.
Agent's registration No. with the Office

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Shrikant Kulkarni,
No. 39, Lake City, K. G. Halli,
Bangalore 560 076, Karnataka State, India.

This person is:
- [ ] applicant only
- [X] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

Applicant's registration No. with the Office

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State *(that is, country)* of residence: IN

This person is applicant for the purposes of:
- [ ] all designated States
- [ ] all designated States except the United States of America
- [X] the United States of America only
- [ ] the States indicated in the Supplemental Box

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Suneet Aurora,
Pharmed Medicare Private Limited,
Pharmed Gardens, Whitefield Road,
Bangalore - 560 048,
Karnataka State, India.

This person is:
- [ ] applicant only
- [X] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

Applicant's registration No. with the Office

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State *(that is, country)* of residence: IN

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- [ ] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

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State *(that is, country)* of residence:

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- [ ] all designated States
- [ ] all designated States except the United States of America
- [ ] the United States of America only
- [ ] the States indicated in the Supplemental Box

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- [ ] applicant only
- [ ] applicant and inventor
- [ ] inventor only *(If this check-box is marked, do not fill in below.)*

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State *(that is, country)* of residence:

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- [ ] all designated States except the United States of America
- [ ] the United States of America only
- [ ] the States indicated in the Supplemental Box

- [ ] Further applicants and/or (further) inventors are indicated on another continuation sheet.

See Notes to the request form

Box No. VIII (ii) DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT

The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (ii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51*bis*.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

in relation to this international application,

Pharmed Medicare Private Limited is entitled to apply for and be granted a patent by virtue of the following:

Pharmed Medicare Private Limited is entitled as employers of the Inventors, namely:

1. Rakesh Ratnam
2. Shrikant Kulkarni
3. Suneet Aurora

This declaration is made for the purposes of all designations, except the designation of the United States of America.

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)".

Box No. VIII (iv) DECLARATION: INVENTORSHIP (only for the purposes of the designation of the United States of America)

The declaration must conform to the following standardized wording provided for in Section 214; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iv). If this Box is not used, this sheet should not be included in the request.

Declaration of inventorship (Rules 4.17(iv) and 51*bis*.1(a)(iv)) for the purposes of the designation of the United States of America:

I hereby declare that I believe I am the original, first and sole (if only one inventor is listed below) or joint (if more than one inventor is listed below) inventor of the subject matter which is claimed and for which a patent is sought.

This declaration is directed to the international application of which it forms a part (if filing declaration with application).

This declaration is directed to international application No. PCT/..... (if furnishing declaration pursuant to Rule 26*ter*).

I hereby declare that my residence, mailing address, and citizenship are as stated next to my name.

I hereby state that I have reviewed and understand the contents of the above-identified international application, including the claims of said application. I have identified in the request of said application, in compliance with PCT Rule 4.10, any claim to foreign priority, and I have identified below, under the heading "Prior Applications," by application number, country or Member of the World Trade Organization, day, month and year of filing, any application for a patent or inventor's certificate filed in a country other than the United States of America, including any PCT international application designating at least one country other than the United States of America, having a filing date before that of the application on which foreign priority is claimed.

Prior Applications: .....

I hereby acknowledge the duty to disclose information that is known by me to be material to patentability as defined by 37 C.F.R. § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the PCT international filing date of the continuation-in-part application.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name: Rakesh Ratnam,

Residence: BANGALORE, INDIA.
(city and either US state, if applicable, or country)

Mailing Address: No.488, 2/B, 39th Cross, 8th Main Jayanagar, Bangalore -560 082, Karnataka State, India.

Citizenship: INDIAN

Inventor's Signature: .....
(if not contained in the request, or if declaration is corrected or added under Rule 26*ter* after the filing of the international application. The signature must be that of the inventor, not that of the agent)

Date: 16/03/04
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26*ter* after the filing of the international application)

Name: Shrikant Kulkarni

Residence: BANGALORE, INDIA.
(city and either US state, if applicable, or country)

Mailing Address: No. 39, Lake City, K.C. Halli, Bangalore - 560076, Karnataka State, India

Citizenship: INDIAN

Inventor's Signature: .....
(if not contained in the request, or if declaration is corrected or added under Rule 26*ter* after the filing of the international application. The signature must be that of the inventor, not that of the agent)

Date: 16/3/04
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26*ter* after the filing of the international application)

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (iv)".

Box No. VIII (v) DECLARATION: NON-PREJUDICIAL DISCLOSURES OR EXCEPTIONS TO LACK OF NOVELTY

The declaration must conform to the standardized wording provided for in Section 215; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (v). If this Box is not used, this sheet should not be included in the request.

Declaration as to non-prejudicial disclosures or exceptions to lack of novelty (Rules 4.17(v) and 51*bis*.1(a)(v)):

Name: Suneet Aurora
Residence: Bangalore, INDIA.
Mailing Address: Pharmed Medicare Private Limited, Pharmed Gardens, Whitefield Road, Bangalore-560 048, Karntaka State, India.
Citizenship: INDIAN

Inventor's Signature:

Date: March 16th 2004

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (v)".

Box No. IX CHECK LIST; LANGUAGE OF FILING

This international application contains:

(a) In paper form, the following number of sheets:

request (including declaration sheets)	6 AA 7 A
description (excluding sequence listing and/or tables related thereto)	8
claims	3
abstract	1
drawings	6
Sub-total number of sheets	
sequence listing	
tables related thereto	
(for both, actual number of sheets if filed in paper form, whether or not also filed in computer readable form; see (c) below)	
Total number of sheets	24 25 A

(b) ☐ **only in computer readable form** (Section 801(a)(i))
- (i) ☐ sequence listing
- (ii) ☐ tables related thereto

(c) ☐ **also in computer readable form** (Section 801(a)(ii))
- (i) ☐ sequence listing
- (ii) ☐ tables related thereto

Type and number of carriers (diskette, CD-ROM, CD-R or other) on which are contained the
- ☐ sequence listing: .....
- ☐ tables related thereto: .....

(additional copies to be indicated under items 9(ii) and/or 10(ii), in right column)

This international application is accompanied by the following item(s) *(mark the applicable check-boxes below and indicate in right column the number of each item)*:

	Number of items
1. ☐ fee calculation sheet	
2. ☐ original separate power of attorney	1
3. ☐ original general power of attorney	
4. ☒ copy of general power of attorney; reference number, if any:	
5. ☐ statement explaining lack of signature	
6. ☒ priority document(s) identified in Box No. VI as item(s):	
7. ☐ translation of international application into *(language)*:	
8. ☐ separate indications concerning deposited microorganism or other biological material	
9. ☐ sequence listing in computer readable form *(indicate type and number of carriers)*	
(i) ☐ copy submitted for the purposes of international search under Rule 13*ter* only (and not as part of the international application)	
(ii) ☐ *(only where check-box (b)(i) or (c)(i) is marked in left column)* additional copies including, where applicable, the copy for the purposes of international search under Rule 13*ter*	
(iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column	
10. ☐ tables in computer readable form related to sequence listing *(indicate type and number of carriers)*	
(i) ☐ copy submitted for the purposes of international search under Section 802(b-*quater*) only (and not as part of the international application)	
(ii) ☐ *(only where check-box (b)(ii) or (c)(ii) is marked in left column)* additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-*quater*)	
(iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column	
11. ☐ other *(specify)*:	

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: ENGLISH

Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Aerrathan
MRS. VAIDYANATHAN ALAMELU

For receiving Office use only

1. Date of actual receipt of the purported international application: 19 MARCH 2004 (19.03.04)
2. Drawings: ☐ received: ☐ not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:
4. Date of timely receipt of the required corrections under PCT Article 11(2):
5. International Searching Authority (if two or more are competent): ISA /
6. ☐ Transmittal of search copy delayed until search fee is paid

For International Bureau use only

Date of receipt of the record copy by the International Bureau:

See Notes to the request form

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PCT/ I NO 4/000 64

24 MARCH 2004

Box No. V DESIGNATIONS

The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents.

However,

☐ DE Germany is not designated for any kind of national protection

☐ KR Republic of Korea is not designated for any kind of national protection

☐ RU Russian Federation is not designated for any kind of national protection

(The check-boxes above may be used to exclude (irrevocably) the designations concerned in order to avoid the ceasing of the effect, under the national law, of an earlier national application from which priority is claimed. See the Notes to Box No. V as to the consequences of such national law provisions in these and certain other States.)

Box No. VI PRIORITY CLAIM

The priority of the following earlier application(s) is hereby claimed:

Filing date of earlier application *(day/month/year)*	Number of earlier application	Where earlier application is: national application: country or Member of WTO	regional application:* regional Office	international application: receiving Office
item (1)				
item (2)				
item (3)				

☐ Further priority claims are indicated in the Supplemental Box.

The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of this international application is the receiving Office)* identified above as:

☐ all items ☐ item (1) ☐ item (2) ☐ item (3) ☐ other, see Supplemental Box

* *Where the earlier application is an ARIPO application, indicate at least one country party to the Paris Convention for the Protection of Industrial Property or one Member of the World Trade Organization for which that earlier application was filed (Rule 4.10(b)(ii)):* ....

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):*

ISA / AT

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority):*

Date *(day/month/year)* Number Country *(or regional Office)*

Box No. VIII DECLARATIONS

The following **declarations** are contained in Boxes Nos. VIII (i) to (v) *(mark the applicable check-boxes below and indicate in the right column the number of each type of declaration)*:		Number of declarations
☐ Box No. VIII (i)	Declaration as to the identity of the inventor	:
☒ Box No. VIII (ii)	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	:
☒ Box No. VIII (iii)	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	:
☒ Box No. VIII (iv)	Declaration of inventorship (only for the purposes of the designation of the United States of America)	:
☐ Box No. VIII (v)	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	:

See Notes to the request form

SUBSTITUTE SHEET

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01 APRIL 2004

PCT

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

PCT / IN04/000064
International Application No.

19 MARCH 2004 (19.03.04)
International Filing Date

THE PATENT OFFICE, (INDIA)
PCT INTERNATIONAL APPLICATION
Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference *(if desired) (12 characters maximum)* IN/PA-62

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Telephone No. 91-80-28410158

Facsimile No. 91-80-28410177

Teleprinter No.

Applicant's registration No. with the Office

State *(that is, country)* of nationality: IN

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This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

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Basaveshwaranagar,
Bangalore - 560 079, Karnataka, India.

Telephone No. 91-80-23235021

Facsimile No. 91-80-23235854

Teleprinter No.

Agent's registration No. with the Office

☐ **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

SUBSTITUTE SHEET

Sheet No. 2

Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)*

Shrikant Kulkarni,
No. 39, Lake City, K. G. Halli,
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- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

Applicant's registration No. with the Office

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- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

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- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

Applicant's registration No. with the Office

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State *(that is, country)* of residence:

This person is applicant for the purposes of:
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- ☐ applicant and inventor
- ☐ inventor only *(If this check-box is marked, do not fill in below.)*

Applicant's registration No. with the Office

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This person is applicant for the purposes of:
- ☐ all designated States
- ☐ all designated States except the United States of America
- ☐ the United States of America only
- ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

Form PCT/RO/101 (continuation sheet) (January 2004) *See Notes to the request form*

SUBSTITUTE SHEET

Box No. V DESIGNATIONS

The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents.

However,

☐ DE Germany is not designated for any kind of national protection

☐ KR Republic of Korea is not designated for any kind of national protection

☐ RU Russian Federation is not designated for any kind of national protection

(The check-boxes above may be used to exclude (irrevocably) the designations concerned in order to avoid the ceasing of the effect, under the national law, of an earlier national application from which priority is claimed. See the Notes to Box No. V as to the consequences of such national law provisions in these and certain other States.)

Box No. VI PRIORITY CLAIM

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Filing date of earlier application *(day/month/year)*	Number of earlier application	Where earlier application is:		
		national application: country or Member of WTO	regional application:* regional Office	international application: receiving Office
item (1)				
item (2)				
item (3)				

☐ Further priority claims are indicated in the Supplemental Box.

The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) *(only if the earlier application was filed with the Office which for the purposes of this international application is the receiving Office)* identified above as:

☐ all items ☐ item (1) ☐ item (2) ☐ item (3) ☐ other, see Supplemental Box

* *Where the earlier application is an ARIPO application, indicate at least one country party to the Paris Convention for the Protection of Industrial Property or one Member of the World Trade Organization for which that earlier application was filed (Rule 4.10(b)(ii)):* ....

.....

Box No. VII INTERNATIONAL SEARCHING AUTHORITY

Choice of International Searching Authority (ISA) *(if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):*

ISA / AT.....

Request to use results of earlier search; reference to that search *(if an earlier search has been carried out by or requested from the International Searching Authority):*

Date *(day/month/year)* Number Country *(or regional Office)*

Box No. VIII DECLARATIONS

The following **declarations** are contained in Boxes Nos. VIII (i) to (v) *(mark the applicable check-boxes below and indicate in the right column the number of each type of declaration)*:		Number of declarations
☐ Box No. VIII (i)	Declaration as to the identity of the inventor	:
☒ Box No. VIII (ii)	Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent	:
☐ Box No. VIII (iii)	Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application	:
☒ Box No. VIII (iv)	Declaration of inventorship (only for the purposes of the designation of the United States of America)	:
☐ Box No. VIII (v)	Declaration as to non-prejudicial disclosures or exceptions to lack of novelty	:

See Notes to the request form

Box No. VIII (ii) DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT

The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (ii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51*bis*.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

in relation to this international application,

Pharmed Medicare Private Limited is entitled to apply for and be granted a patent by virtue of the following:

Pharmed Medicare Private Limited is entitled as employers of the Inventors, namely:

1. Rakesh Ratnam
2. Shrikant Kulkarni
3. Suneet Aurora

This declaration is made for the purposes of all designations, except the designation of the United States of America.

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)".

Box No. VIII (iv) DECLARATION: INVENTORSHIP (only for the purposes of the designation of the United States of America)

The declaration must conform to the following standardized wording provided for in Section 214; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iv). If this Box is not used, this sheet should not be included in the request.

Declaration of inventorship (Rules 4.17(iv) and 51*bis*.1(a)(iv)) for the purposes of the designation of the United States of America:

I hereby declare that I believe I am the original, first and sole (if only one inventor is listed below) or joint (if more than one inventor is listed below) inventor of the subject matter which is claimed and for which a patent is sought.

This declaration is directed to the international application of which it forms a part (if filing declaration with application).

This declaration is directed to international application No. PCT/..... (if furnishing declaration pursuant to Rule 26*ter*).

I hereby declare that my residence, mailing address, and citizenship are as stated next to my name,

I hereby state that I have reviewed and understand the contents of the above-identified international application, including the claims of said application. I have identified in the request of said application, in compliance with PCT Rule 4.10, any claim to foreign priority, and I have identified below, under the heading "Prior Applications," by application number, country or Member of the World Trade Organization, day, month and year of filing, any application for a patent or inventor's certificate filed in a country other than the United States of America, including any PCT international application designating at least one country other than the United States of America, having a filing date before that of the application on which foreign priority is claimed.

Prior Applications: .....

I hereby acknowledge the duty to disclose information that is known by me to be material to patentability as defined by 37 C.F.R. § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the PCT international filing date of the continuation-in-part application.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name: Rakesh Ratnam,

Residence: BANGALORE, INDIA.
(city and either US state, if applicable, or country)

Mailing Address: No.488, 2/B, 39th Cross, 8th Main Jayanagar, Bangalore -560 082, Karnataka State, India.

Citizenship: INDIAN

Inventor's Signature: .....
(if not contained in the request, or if declaration is corrected or added under Rule 26*ter* after the filing of the international application. The signature must be that of the inventor, not that of the agent)

Date: 16.03.2004
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26*ter* after the filing of the international application)

Name: Shrikant Kulkarni

Residence: BANGALORE, INDIA.
(city and either US state, if applicable, or country)

Mailing Address: No. 39, Lake City, K.C. Halli, Bangalore - 560076, Karnataka State, India

Citizenship: INDIAN

Inventor's Signature: .....
(if not contained in the request, or if declaration is corrected or added under Rule 26*ter* after the filing of the international application. The signature must be that of the inventor, not that of the agent)

Date: 16.03.2004
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26*ter* after the filing of the international application)

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (iv)".

SUBSTITUTE SHEET

Continuation of Box No. VIII (iv) **DECLARATION**

If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII ..." (indicate the item number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in respect of two or more declarations, a separate continuation box must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.

Name: Suneet Aurora
Residence: Bangalore, INDIA.
Mailing Address: Pharmed Medicare Private Limited, Pharmed Gardens, Whitefield Road, Bangalore-560 048, Karntaka State, India.
Citizenship: INDIAN

Inventor's Signature:

Date: 16.03.2004

Box No. IX CHECK LIST; LANGUAGE OF FILING

This international application contains:

(a) **in paper form, the following number of sheets:**

request (including declaration sheets)	7
description (excluding sequence listing and/or tables related thereto)	8
claims	3
abstract	1
drawings	6
Sub-total number of sheets	
sequence listing	
tables related thereto	
(for both, actual number of sheets if filed in paper form, whether or not also filed in computer readable form: see (c) below)	
Total number of sheets	25

(b) ☐ **only in computer readable form (Section 801(a)(i))**
- (i) ☐ sequence listing
- (ii) ☐ tables related thereto

(c) ☐ **also in computer readable form (Section 801(a)(ii))**
- (i) ☐ sequence listing
- (ii) ☐ tables related thereto

Type and number of carriers (diskette, CD-ROM, CD-R or other) on which are contained the
- ☐ sequence listing: .....
- ☐ tables related thereto: .....

(additional copies to be indicated under items 9(ii) and/or 10(ii), in right column)

This international application is accompanied by the following item(s) *(mark the applicable check-boxes below and indicate in right column the number of each item)*:

	Number of items
1. ☐ fee calculation sheet	
2. ☒ original separate power of attorney	1
3. ☐ original general power of attorney	
4. ☐ copy of general power of attorney; reference number, if any:	
5. ☐ statement explaining lack of signature	
6. ☐ priority document(s) identified in Box No. VI as item(s):	
7. ☐ translation of international application into *(language)*:	
8. ☐ separate indications concerning deposited microorganism or other biological material	
9. ☐ sequence listing in computer readable form *(indicate type and number of carriers)*	
(i) ☐ copy submitted for the purposes of international search under Rule 13*ter* only (and not as part of the international application)	
(ii) ☐ *(only where check-box (b)(i) or (c)(i) is marked in left column)* additional copies including, where applicable, the copy for the purposes of international search under Rule 13*ter*	
(iii) ☐ together with relevant statement as to the identity of the copy or copies with the sequence listing mentioned in left column	
10. ☐ tables in computer readable form related to sequence listing *(indicate type and number of carriers)*	
(i) ☐ copy submitted for the purposes of international search under Section 802(b-*quater*) only (and not as part of the international application)	
(ii) ☐ *(only where check-box (b)(ii) or (c)(ii) is marked in left column)* additional copies including, where applicable, the copy for the purposes of international search under Section 802(b-*quater*)	
(iii) ☐ together with relevant statement as to the identity of the copy or copies with the tables mentioned in left column	
11. ☐ other *(specify)*:	

Figure of the drawings which should accompany the abstract:

Language of filing of the international application: ENGLISH

Box No. X SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Alamelu

MRS. VAIDYANATHAN ALAMELU

For receiving Office use only

1. **Date of actual receipt of the purported international application:** 19 MARCH 2004 (19.03.04)

2. **Drawings:** ☐ received: ☐ not received:

3. **Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:**

4. **Date of timely receipt of the required corrections under PCT Article 11(2):**

5. **International Searching Authority (if two or more are competent):** ISA/

6. ☐ **Transmittal of search copy delayed until search fee is paid**

For International Bureau use only

Date of receipt of the record copy by the International Bureau:

SUBSTITUTE SHEET

AN IMPROVED PROCESS FOR PRODUCING CHLORINATED SUCROSE

This invention relates to a process for producing chlorinated sucrose, mainly 1',6'-Dichloro-1',6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy Galactopyranoside by using an agitated thin film dryer during the intermediate step of solvent stripping and deacetylation. And there after obtaining a pure dried form of high purity molecule.

BACKGROUND OF THE INVENTION:

Chloro derivatives derived from sugars, exhibit the organoleptic properties with a very high degree of sweetness compared to the parent sugar. One such chloro sugar prepared from sucrose is 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside. It is a well-known sweetener used widely, including in food and food preparations. Various synthetic routes for the production of 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-Galactopyranoside are reported in literature, for e.g. Fair Clough et al, carbohydrate research 40(1975) 285-298 Mufti et.,al., US Patent No. 4,380,476 and British Patent No. 1543167.

Following major challenges are faced during the preparation:

1. Introduction of chlorine atoms in positions 4, 1' and 6' of sucrose molecule.
2. Isolation of the pure 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside from quenched reaction mixture.
3. Isolation of solids from reaction mixture.
4. Extraction of the product from solids and crystallization of the product.
5. Isolation of pure product from the solid extracts.

Sucrose –6 acetate is chlorinated using Vilsmeyer Haack reaction. The bottleneck was the isolation of pure 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside from the quenched chlorinated mass. Procedures for this

also have been reported in patent literature. The major concern here was the complete removal of the solvent from the reaction mixture before deacetylation of the 1', 6'-Dichloro-1',6'-Dideoxy-β-D-Fructo-Furanosyl-4-chloro-4-Deoxy-6-acetoxy-α-D-Galactopyranoside that is the main intermediate in almost all the reported process. The solvents used in this process may be dimethyl sulphoxide, dimethyl formamide, pyridine, hexane, or cyclohexane.

The desired product is produced from Sucrose 6 Acetate. During the process, a major problem is removal of solvents and isolation of the solids from the reaction mixture at low temperature without causing appreciable degradation. Earlier the solvents removal was done by steam distillation. The suggested steam distillation operation, in the prior art, is highly energy consuming and the volume of the mass increases to 4 - 5 times of the original volume. Isolation of the product from this reaction mass is again a time consuming and tedious process.

BRIEF SUMMARY OF THE INVENTION:

The chlorination of sucrose-6-acetate was carried out by using Vilsmeier- Haack reagent, which was generated by adding phosphorus oxy chloride or phosphorous penta chloride to a highly polar solvent like dimethyl formamide. The reaction mass was quenched by sodium hydroxide solution in ice and the deacetylation of 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-6-acetoxy-α-D-Galactopyranoside-6-acetate was carried out by adjusting the pH of the quenched mass with agitation. .

The quenched mass was fed into the agitated thin film dryer of appropriate cross section area having a flanged scrapper type rotor. The description of the agitated thin film dryer is discussed in details in this document. The solids obtained from the dryer contain essentially the product as well as the inorganic salts such as chlorides and phosphates. Three different approaches are attempted to isolate the pure product from the solids obtained from the dryer.

a) Extracting the solids with an appropriate solvent e.g. an alcohol and purification of the crude mass by column chromatography followed by crystallization using ATFD or spray dryer.

b) Direct column chromatography of the solids over a packed column using for example silica or alumina.

c) Dissolving the solids in water and then extracting the aqueous solution with Organic solvents which are not miscible with water like dichloromethane, ethyl acetate or toluene. Stripping of the solvent to get a mass which is finally crystallized to afford pure 1', 6'-Dichloro-1',6' Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-alactopyranoside.

Removal of solvent from the quenched mass of the reaction mixture by ATFD is being reported for the first time in this type obtaining a dry or semidry powder on removal of the solvent is being reported of for the first time reaction. The design of the agitated thin film dryer is such that the whole operation is done at a lower temperature & pressure. This in turn affords better quality product and yield compared to the earlier methods as mentioned in the experimental section of this document.

Given herein below are the short forms used in the specification along with the expansions:

$POCL_3$ stands for Phosphorous oxy-chloride

ATFD stands for Agitated Thin Film Dryer

MEK stands for Methyl Ethyl Ketone

TLC stands for Thin Layer Chromatography

HPLC stands for High Pressure Liquid Chromatography

In the accompanying drawings:

Fig. 1 illustrates the reaction scheme for the preparation of 1', 6'-Dichloro-1', 6'-Dideoxy -β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside;

Fig. 2 illustrates the agitated thin film dryer used in the process of the present invention;

Fig. 3 is the flow sheet of the agitated thin film dryer;

Fig. 4 is the flow chart of the process of the present invention;

Fig. 5 is the IR Report of the product of the present invention; and

Fig. 6 is the HPLC Chromatogram of the product of the present invention.

DETAILED DESCRIPTION OF THE INVENTION:

The process is an useful improvement in the manufacture of 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside. The reaction scheme involved in the manufacture of product from sucrose-6- acetate is given in fig. 1 of the accompanying drawings.

The sucrose-6-acetate is chlorinated to give 1',6'-Dichloro-1',6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside.

The Vilsemeyer reagent is prepared from Phosphorus Oxy Chloride ($POCl_3$) or phosphorus penta chloride (PCl_5). The sucrose-6-acetate is added to Vilsmeyer Reagent at 5°-10°C. After completion of the reaction, the reaction mass is heated to 80° to 100°C and preferably between 90°-95°C and maintained for ½ -1hr and then the temperature is raised to 110° to 135°C and preferably to 120°-125°C and maintained for 3-5 hours. There after the reaction mass is cooled to room temperature and quenched into an inorganic basic solution like alkali hydroxide or carbonate solution, for example Sodium hydroxide solution, containing ice or chilled using brine or other cooling agent solution in the jacket of the reactor. The temperature during the quenching operation is maximum 30°C - 35°C. The pH is adjusted to 7.5 to 14 and preferably 10-13 by 15-20% of alkali hydroxide solution in water. At this pH the mass is stirred to complete the deacetylation.

The quenched mass comprises of inorganic salts, chlorinated sucrose-6-acetate, solvent and water. This is fed into ATFD for the removal of solvent and water. Schematic Diagram of ATFD is shown in Fig. 2 of the drawings.

The detailed process of ATFD is illustrated as follows.

Feed of the quenched mass was cooled to 5 to 10°C degree in the feed tank by circulating a brine solution. A pump was used to lift the feed from feed tank to the dryer. The ATFD is a vertical dryer with area of cross section 0.25 to 0.35m^2. The feed enters tangentially and spreads along the inside surface of the shell in to a thin film. The rotor blades are hinged, the hinged rotor blades keep the film under intense agitation preventing any scale formation. The speed of the rotor was 1000 to 1500rpm.The film progressively passes through different phases like liquid, slurry, paste, wet powder and finely powder of desired dryness, it is collected in a powder receiver.

The vapor flows countercurrent to the solids and was removed from the top of the dryer. Distillate was collected from the condenser and solids are obtained from the dryer. The distillate contains solvent and water. The distillate was subjected to fractional distillation, about 70-80% of solvent was recovered based on the input of solvent.

EXPERIMENTAL DETAILS:

CHLORINATION OF SUCROSE-6-ACETATE:

100grm of sucrose 6-acetate is mixed with 200 ml of fresh solvent such as hexane, cyclohexane, pyridine, dimethyl formamide, and others, and particularly dimethyl formamide and Chlorination undertaken in a 3 liter 3 neck round bottom flask. 500 ml of the solvent is charged. Thereafter, the solvent is cooled with stirring to 0 to –5°C, to this reaction mass 166 ml of phosphorous oxy chloride (273.9gm) is added below 0°C. To this 100grm of sucrose 6-acetate in solvent is added below 10°C. Thereafter, the reaction mass is stirred at 20-25°C for ½-1hr.The temperature is raised to 70-100°C and preferably 80- 90°C and maintained for 1 to 2 hours. Afterwards the temperature is raised to 110 to 130°C and preferably 120- 122°C and maintained for 3 to 5 hrs. The reaction mass is cooled to 40°-45°C, the reaction mass is added to a solution containing 220grm sodium hydroxide solution, 220grm water and 1000grm ice. The pH is adjusted to 7.5 to 14 and preferably 10-13 and stirred for 3-5 hour at room temperature. The mass is filtered. The

residue was washed with 20 ml of the solvent and then the washings are combined with the main filtrate. The resultant mass is fed into ATFD for solvent removal.

ATFD REMOVAL OF SOLVENT:

The quenched mass approximate volume of 2-2.3 lit and pH is adjusted to 10-13 and fed to ATFD with the following parameters.

Area of ATFD=		0.20 - 0.50 m^2
Feed rate	=	7-10kg/hr
Pressure	=	2 - 10mmHg.
Jacket temp	=	70 – 100°C

Feed of 3 to 5Kg was cooled down to 5-10°C in the feed tank by circulating the brine solution. pH of the feed was maintained at 7.5 to 14 and preferably between 10-13; the pump was fitted to lift the feed from feed tank to the dryer. The dryer is a vertical dryer with area of cross section 0.25-0.35 Sq.m. The feed enters tangentially and spreads along the inside surface of the shell in to a thin film. The rotor blades are hinged, the hinged rotor blades keep the film under intense agitation preventing any scale formation. The speed of the rotor was 1000-1500rpm. Temperature was maintained around 70-100°C in the jacket by circulating hot water taking inlet from bottom, outlet through the top. The film progressively passes through different phases like liquid, slurry, paste, wet powder and finely powder of desired dryness. This is collected in a powder receiver.

The vapor flows countercurrent to the solids and was removed from the top of the dryer; these vapors are condensed in the condenser. Distillate was collected from the condenser, solids are obtained from the dryer. The distillate contains solvent and water. The distillate was subjected to fractional distillation, about 70-80% of solvent was recovered based on the input of solvent.

PRODUCT ISOLATION:

The mass obtained from the ATFD is subjected to solvent extraction. The solvent used may be any organic solvent, including but not limited to, ethyl acetate, methanol, methyl ethyl ketone, and acetone. The preferred solvent may be ethyl acetate. The solvent extracted mass is distilled in the rotary evaporator at low temperature. The syrup obtained is mixed with an appropriate column chromatography adsorbent like silica or Alumina and run through column chromatography. The adsorbing agent could be any known column packing preferably Alumina or silica. The preferable solvents for desorbption are ethyl acetate, mixture of toluene and methanol, mixture of methanol and ethyl acetate, mixture of methanol and dichloromethane. The eluted fractions are collected in different receivers based on TLC showings. The fractions showing single spot on the TLC are collected separately. The solvent from this fraction was evaporated to provide a thick syrup. The thick syrup is subjected to purification. Crude product obtained showed by TLC to have a high concentration of the desired product. This was subjected to crystallization.

PURIFICATION OF THE PRODUCT:

The syrup obtained from the isolation stage is mixed with 2-5 volumes of a polar solvent, example methanol, and the mass is mixed with charcoal at 50 to 60°C. The resulting mixture is filtered through a hyflo adsorbent and the hyflo bed is washed with 1 volume of the same solvent. Then, the filtered mass is distilled at low temperature to remove 90% solvent and to this 3-5 volumes of mass of ethyl acetate is added and mixed well. The mass is distilled at low temperature to remove 2-4 volumes of ethyl acetate resulting in a liquid product concentrate. The desired dry product was obtained from the liquid concentrate by three methods,

a. Conventional crystallization, as reported as in the earlier patents cited in this document.

b. Feeding liquid product concentrate to ATFD to obtain the dry desired pure product. This is being reported for the first time.

c. Spray drying the liquid product concentrate to obtain dry desired pure product. This is being reported for the first time.

In the conventional crystallization method the liquid concentrate was crystallized to obtain solid product. The product was filtered and dried under vaccum at 40 to 50°C. The solids isolated after feeding the concentrate in the ATFD were also found to be identical with the product obtained from the conventional crystallization method. Also the solid obtained after spray drying the liquid concentrate are found to be identical with the desired pure product obtained from the crystallization and ATFD method. The analysis of the solids from all the three methods showed the product purity or content is over 99% (HPLC Fig. 6 and IR Fig. 5 attached). In a separate experiment, the charcolised reaction mass after passing through the hyflo bed but before concentration is taken for further recovery of the product. The product can be isolated directly by feeding the purified charcoalised methanol solution into the ATFD. This also resulted in a desired pure product of high purity.

The solid product could be also isolated by an alternative method. This method comprises of feeding the charcoalised methanol solution obtained after column chromatography directly into the spray dryer to afford the solid product.

CLAIMS:

1. In a process for producing chlorinated sucrose, mainly 1',6'-Dichloro-1',6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside by chlorination of 6-acetyl sucrose in the presence of a solvent such as dimethyl sulfoxide, dimethyl formamide, pyridine, hexane, cyclohexane, deacetylation, removal of solvents used, extraction, purification and crystallization, the improvement comprising the removal of the solvents using an Agitated Thin Film Dryer or Spray Dryer by maintaining the temperature around 70-100°C in the jacket of ATFD and the pH of the feed at 7.5 to 14 by the addition of Alkali solution and optional crystallization of the product by feeding purified charcolised methanol solution into the said dryers, before or after concentration of the solvent mass after the removal of charcoal.

2. A process for producing chlorinated sucrose as claimed in claim 1, wherein the temperature in the dryer being maintained at 70-100°C and preferably at 80°C by circulating hot water in the jacket having inlet from the bottom of the vessel and outlet at the top.

3. A process for producing chlorinated sucrose as claimed in claim 1, wherein agitated thin film dryer is a vertical dryer having a rotor to keep the film formed by the chlorinated mass under intense agitation to prevent scaling and localized heating.

4. A process for producing chlorinated sucrose as claimed in claim 1, wherein the vapor flows countercurrent to the solids and is removed from the top of the dryer.

5. In a process for producing 1', 6'-Dichloro-1', 6'-Dideoxy -β-D-Fructo-Furanosyl-

4-Chloro-4-Deoxy-α-D-Galactopyranoside by chlorination of 6-acetyl sucrose in the presence of a solvent such as dimethyl sulfoxide, dimethyl formamide, pyridine, hexane, cyclohexane, deacetylation, removal of the solvent used, extraction, purification and crystallization, the improvement comprising the removal of the solvent using an agitated thin film dryer having a rotor to keep the film formed by the chlorinated mass under intense agitation to prevent scaling, maintaining the temperature around 70 to 100°C and preferably at about 80°C by circulating hot water in the jacket having inlet from the bottom of the vessel and outlet at the top, the pH of the feed being maintained at 7.5 to 14 and preferably at 10 to 13 by the addition of and optional isolation of the product by feeding purified charcolised methanol solution into the said dryer.

6. A process as claimed in claim 5, wherein the mass obtained from the thin film dryer is subjected to solvent extraction.

7. A process as claimed in claim 6, wherein the solvent used is selected from any organic solvent and particularly from ethyl acetate, methanol, methyl ethyl ketone, dimethyl sulphoxide, dimethyl formamide, pyridine, hexane, or cyclohexane and acetone.

8. A process as claimed in claim 7, wherein the solvent extracted mass is concentrated distilled in a rotary evaporator at low temperature, the syrup obtained is mixed with any adsorbent like silica, alumina and run through column chromatography.

9. A process as claimed in claim 8, wherein the isolated syrup obtained is mixed with two volumes of solvents example methanol and the mass is then treated with charcoal at 50-60°C, filtering the mass followed by distillation at low temperature to remove 90% solvent, thoroughly mixing the product obtained with three

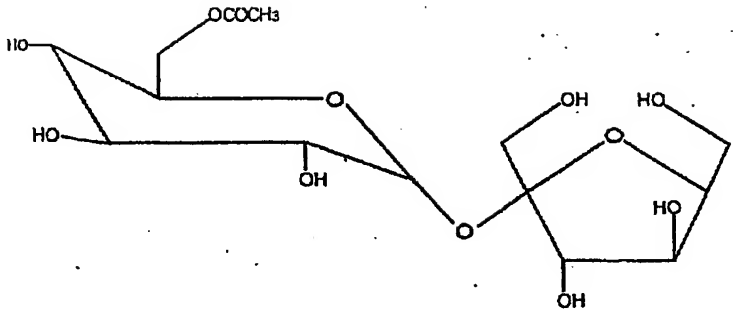
volumes of appropriate solvents, followed by a further distillation at low temperature to remove the solvents and crystallizing the product by known methods or by the said new methods i.e. Agitated Thin Film Drying or Spray Drying.

10. In a process for producing 1', 6'-Dichloro-1', 6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside by chlorination of 6-acetyl sucrose in the presence of a solvent such as dimethyl sulfoxide, dimethyl formamide, pyridine, hexane, cyclohexane, deacetylation, removal of the solvent used, extraction, purification and crystallization, the improvement comprising the removal of the solvent using an agitated thin film dryer having a rotor to keep the film formed by the chlorinated mass under intense agitation to prevent scaling and localized heating maintaining the temperature around 70 to 100°C and preferably at 80°C by circulating hot water in a jacket having inlet from the bottom of the vessel and outlet at the top, the pH of the feed being maintained at 7.5 to 14 and preferably at 10 to 13 by the addition of alkali hydroxide solution and crystallization of the product by feeding purified charcoalised solvent into the dryer.

11. A process as claimed in claim 10, wherein the isolation of the pure crystalline 1',6'-Dichloro-1',6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside is carried out by using spray drying technique.

ABSTRACT

Disclosed herein is a process for producing chlorinated sucrose, mainly 1',6'-Dichloro-1',6'-Dideoxy-β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α-D-Galactopyranoside by chlorination of 6-acetyl sucrose in the presence of a solvent such as dimethyl sulfoxide, dimethyl formamide, pyridine, hexane, cyclohexane, deacetylation, removal of solvents used, extraction, purification and crystallization, the improvement comprising the removal of the solvents using an Agitated Thin Film Dryer or Spray Dryer by maintaining the temperature around 70-100°C in the jacket of ATFD and the pH of the feed at 7.5 to 14 by the addition of Alkali solution and optional crystallization of the product by feeding purified charcolised methanol solution into the said dryers, before or after concentration of the solvent mass after the removal of charcoal.

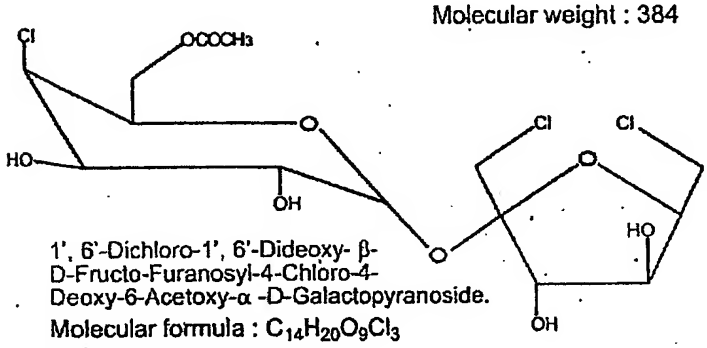


Sucrose - 6 - Acetate

Molecular weight : 384

$POCl_3$ +DMF
115°C

Chlorination

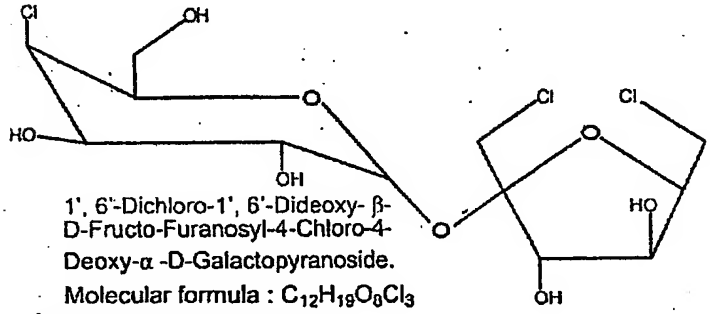


1', 6'-Dichloro-1', 6'-Dideoxy- β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-6-Acetoxy-α -D-Galactopyranoside.

Molecular formula : $C_{14}H_{20}O_9Cl_3$

Molecular weight : 439.5

De Acetylation



1', 6'-Dichloro-1', 6'-Dideoxy- β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α -D-Galactopyranoside.

Molecular formula : $C_{12}H_{19}O_8Cl_3$

Molecular weight : 397.64

FIGURE -1

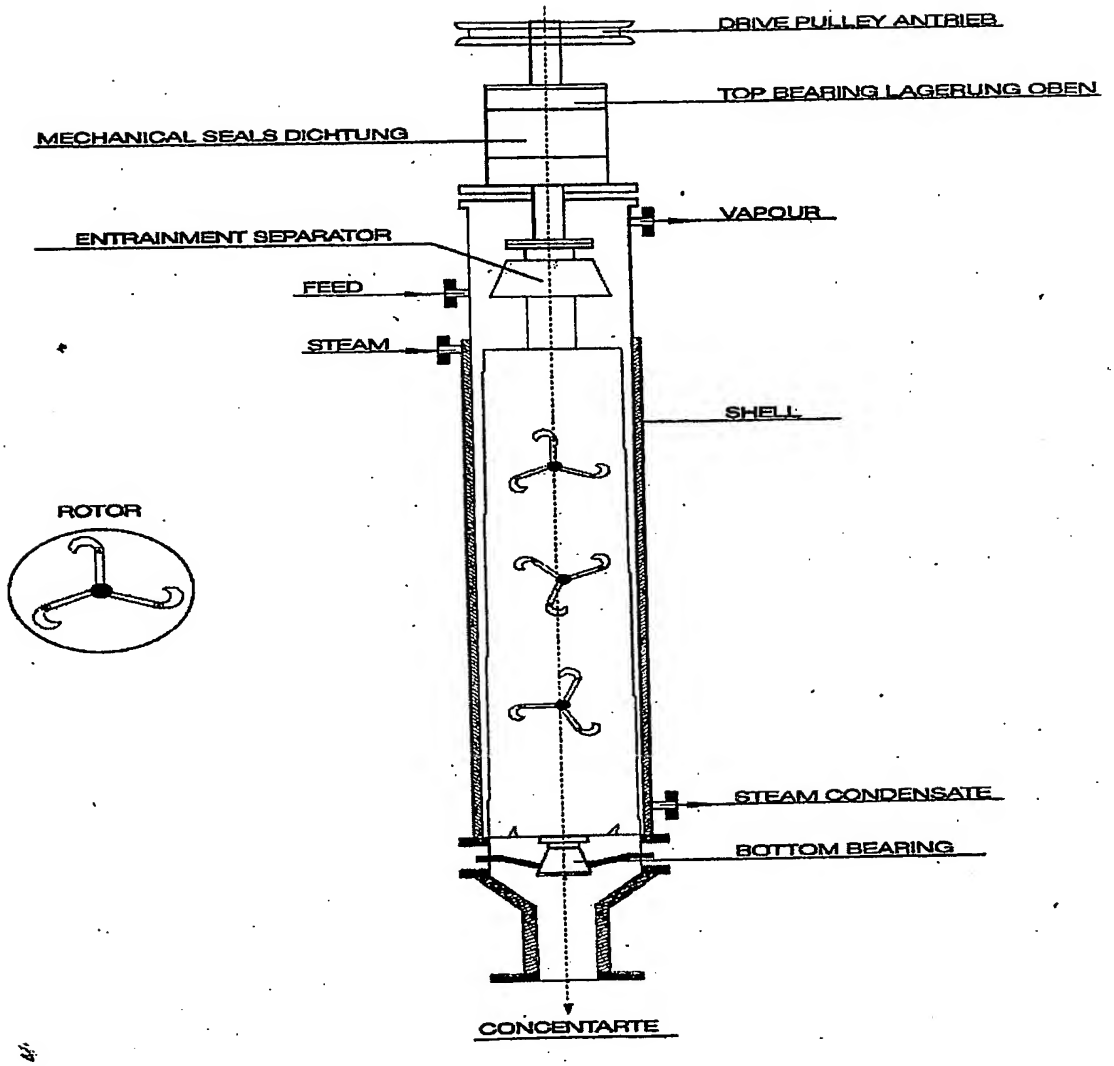

DRIVE PULLEY ANTRIEB
TOP BEARING LAGERUNG OBEN
MECHANICAL SEALS DICHTUNG
VAPOUR
ENTRAINMENT SEPARATOR
FEED
STEAM
SHELL
ROTOR
STEAM CONDENSATE
BOTTOM BEARING
CONCENTARTE

FIGURE-2

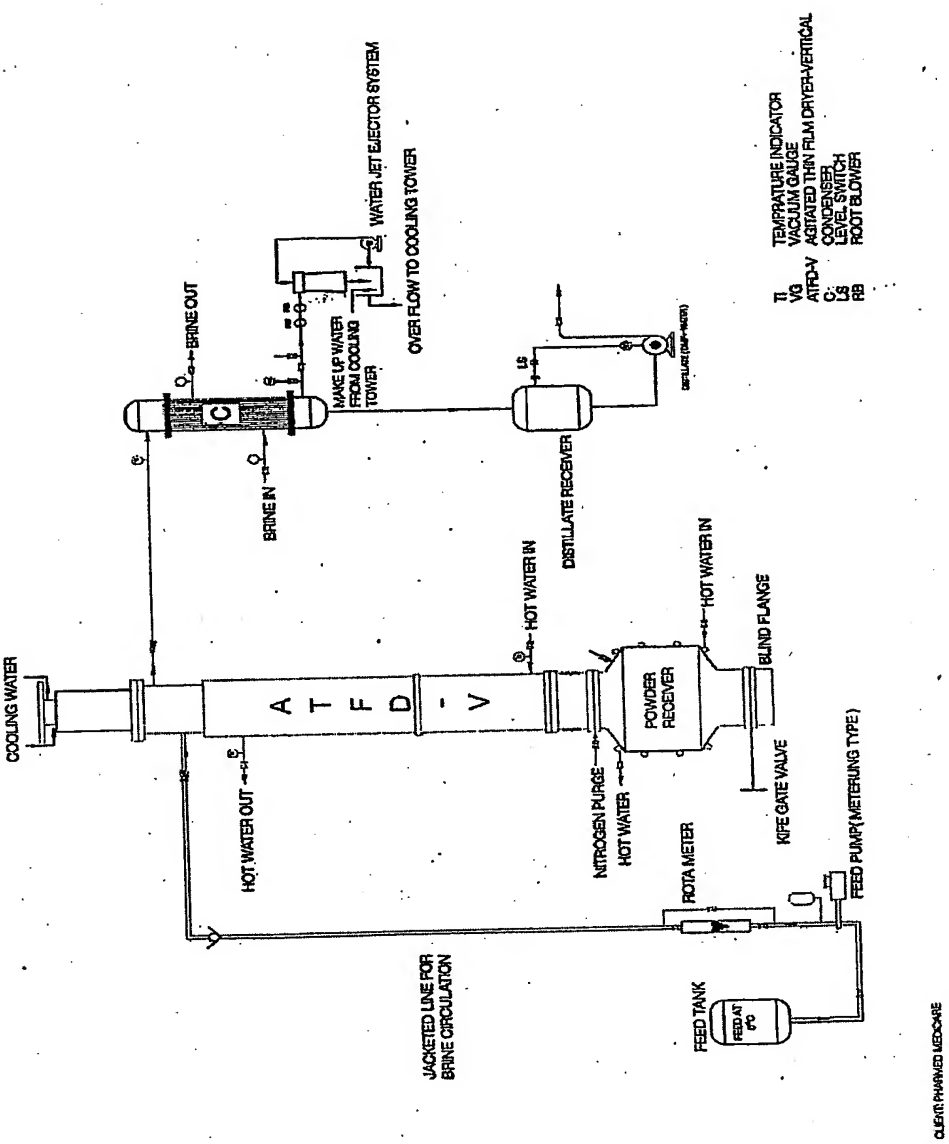

TI TEMPRATURE INDICATOR
VG VACUUM GAUGE
ATFD-V AGITATED THIN FILM DRYER-VERTICAL
C CONDENSER
LS LEVEL SWITCH
RB ROOT BLOWER
WATER JET EJECTOR SYSTEM
OVER FLOW TO COOLING TOWER
MAKE UP WATER FROM COOLING TOWER
BRINE OUT
BRINE IN
DISTILLATE RECEIVER
HOT WATER IN
HOT WATER IN
BLIND FLANGE
COOLING WATER
A T F D - V
POWDER RECEIVER
HOT WATER OUT
NITROGEN PURGE
HOT WATER
ROTA METER
KIPE GATE VALVE
FEED PUMP(METERING TYPE)
JACKETED LINE FOR BRINE CIRCULATION
FEED TANK
FEED AT 6°C
CLIENT: PHARMED MEDICARE

FIGURE-3

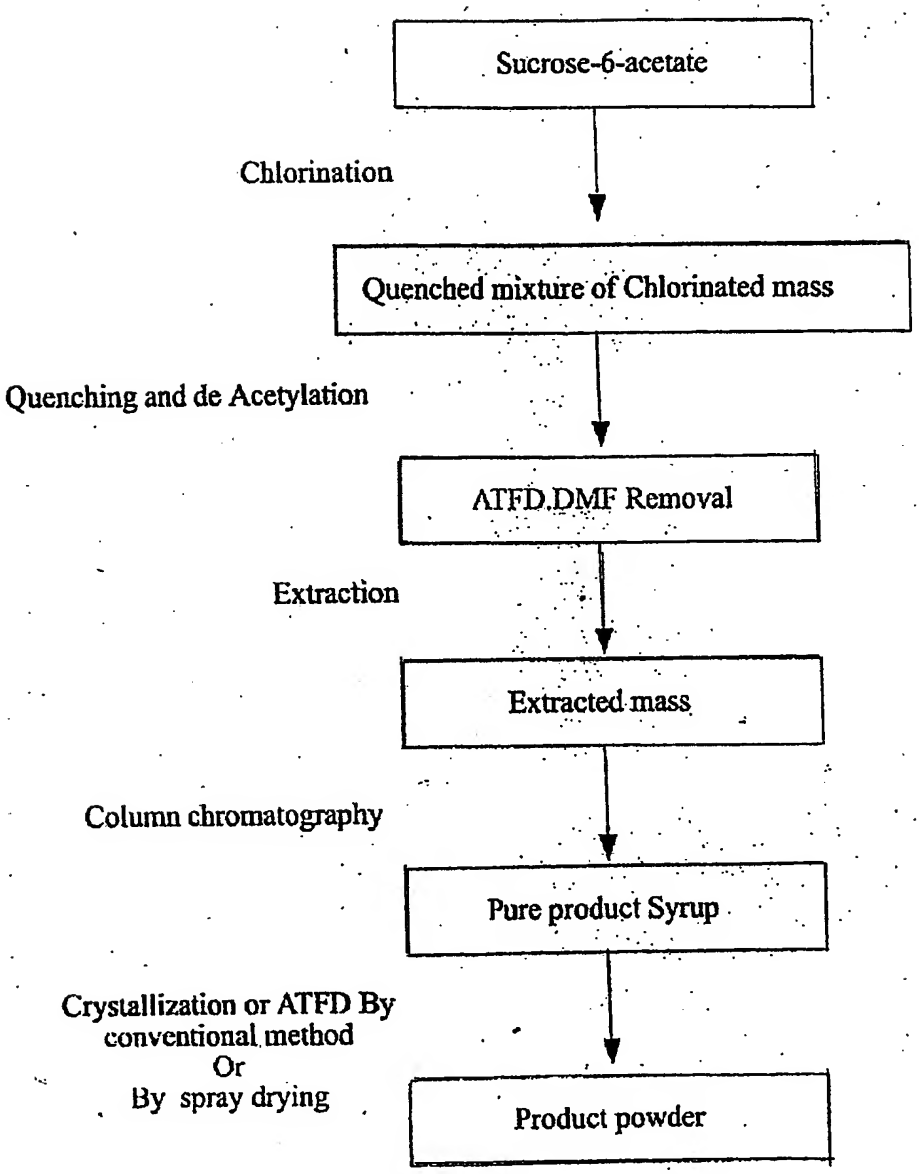

Sucrose-6-acetate
Chlorination
Quenched mixture of Chlorinated mass
Quenching and de Acetylation
ATFD.DMF Removal
Extraction
Extracted mass
Column chromatography
Pure product Syrup
Crystallization or ATFD By conventional method
Or
By spray drying
Product powder

FIGURE -4

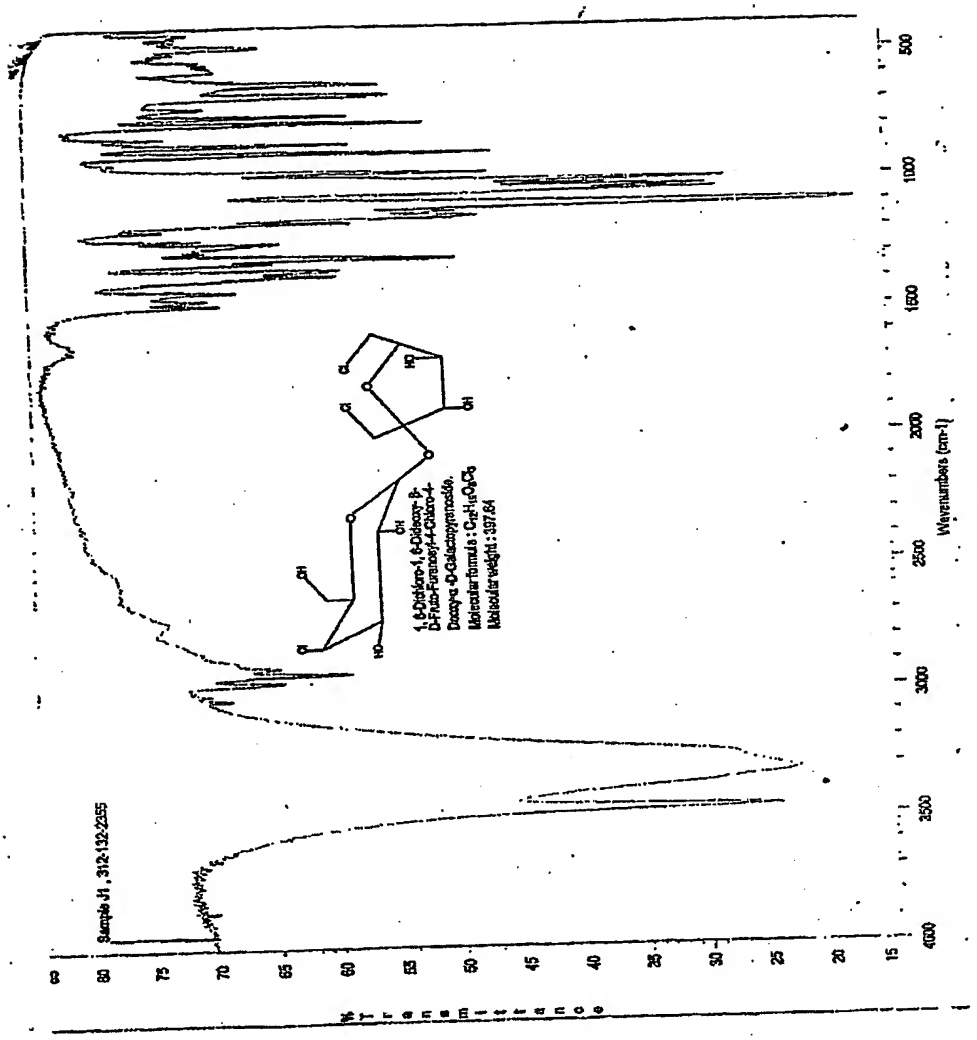
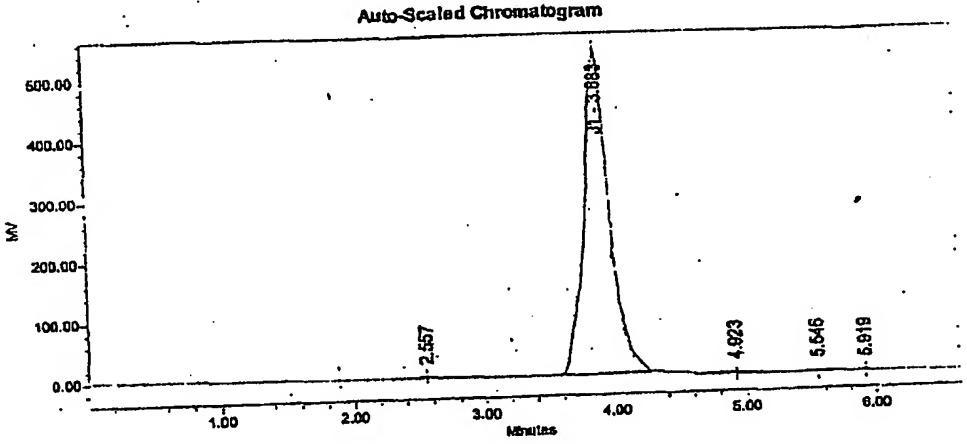

Sample J1 , 312-132-2355
1,6-Dideoxy-1,6-Dideoxy-β-
D-Fructo-Furanosyl-4-Chloro-4-
Deoxy-α-D-Galactopyranoside,
Molecular formula : C12H21ClO9
Molecular weight : 337.84
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
500
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15

FIGURE-5

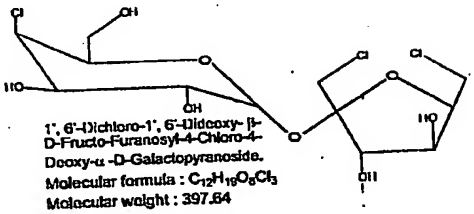
SampleName	1', 6'-Dichloro-1', 6'-Dideoxy- b- D-Fructo-Furanosyl-4-Chloro-4- Deoxy-a -D-Galactopyranoside.
Vial	1
Injection	4
Injection Volume	10.00 ul
Channel	410
Run Time	6.0 Minutes
Sample Type	Sample
Date Acquired	1/9/04 10:10:03 AM
Acq Method Set	PHARMED_MTH
Processing Method	PHARMED PRO
Date Processed	

Auto-Scaled Chromatogram



Peak Results

	Peak Name	RT	Area	Height	% Area	% Height	Int Type
1		2.557	36707	2450	0.51	0.45	Bb
2	J1	3.883	7130944	539348	98.35	98.87	bb
3		4.923	59512	1928	0.82	0.35	BB
4		5.546	9102	833	0.13	0.15	BB
5		5.919	14342	948	0.20	0.17	BB



1', 6'-Dichloro-1', 6'-Dideoxy- β-D-Fructo-Furanosyl-4-Chloro-4-Deoxy-α -D-Galactopyranoside.
Molecular formula : $C_{12}H_{19}O_8Cl_3$
Molecular weight : 397.64

FIGURE - 6